NEUTRON-ACTIVATION ANALYSIS

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REPRINTED FROM
SCIENTIFIC AMERICAN
APRIL 1967
VOL. 216, NO. 4 PP. 68-82

PUBLISHED BY W. H. FREEMAN AND COMPANY 660 MARKET STREET, SAN FRANCISCO, CALIFORNIA 94104

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NEUTRON-ACTIVATION ANALYSIS

This powerful analytic tool is now routinely used in a wide range of disciplines to measure the concentrations of trace elements, often in amounts less than a billionth of a gram

by Werner H. Wahl and Henry H. Kramer

In most physical or chemical investigations impurities are regarded as a nuisance. In recent years, however, experiments in a wide range of fields have been devoted to the painstaking analysis of just such "trace elements." Part of the impetus of this trend has come from the growing realization that minute amounts of certain elements are essential to the metabolism of all living organisms. Accordingly the identification and measurement of trace elements have become indispensable aids to the understanding of many biological processes. The analysis of trace elements also plays an important role in such diverse disciplines as geology, medicine, archaeology, electronics, metallurgy, art history and criminology. Among the techniques devised for measuring the concentration of trace elements perhaps the most sensitive and versatile is neutron-activation analysis.

Reduced to its simplest terms, this method of analysis consists of the following main operations. First a sample of material is bombarded by an intense beam of neutrons. The neutrons interact with the nuclei of atoms in the sample, in many cases to form radioactive nuclei. The sample is then removed from the path of the neutron beam, whereupon the radioactivity of the sample can be measured. The radiation emitted by such "radionuclides" has a characteristic spectrum that can be analyzed to deduce both the elemental constituents of the sample and their respective concentrations. So far variations of this basic technique have been used to detect the presence of some 70 different elements, sometimes in amounts as small as a trillionth (10^{-12}) of a gram! In our laboratory at the Sterling Forest Research Center of the Union Carbide Corporation we have been working for the past few years to improve the sensitivity and extend the usefulness of this powerful analytic tool.

Neutron-activation analysis is one of the many unanticipated dividends of basic research into the nature of the atomic nucleus. The phenomenon of radioactivity—the spontaneous decay of a nucleus accompanied by the emission of radiation—was identified in some natural substances as early as 1898, but it was not until 1933 that Irène and Frédéric Joliot-Curie noted that when they placed pieces of aluminum next to a sample of naturally radioactive polonium, radiation was emitted that did not come from the polonium; moreover, the samples remained radioactive even after the polonium was removed. Chemical analysis showed that the "induced" radioactivity came from a radioisotope, or radioactive species, of phosphorus produced by the disintegration of the aluminum nuclei. The phosphorus decayed at a rate not previously observed for any naturally radioactive substance. This was the first reported observation of an artificial radioisotope.

A few years later the noted chemist Georg von Hevesy, "without having a special reason to do so," exposed a purified salt of the element dysprosium to a stream of neutrons emitted by a radioactive mixture of radium, radon and beryllium. He observed that the irradiated salt displayed "the most intense artificial radioactivity.... This observation induced me to determine the dysprosium content of an yttrium sample by exposing it to a neutron stream." Thus, on an impulse, von Hevesy discovered the principle of neutron-activation analysis.

The hiatus between the discovery of activation analysis and its routine application resulted from the lack of sufficiently intense neutron sources and the inability of early detection devices to resolve the various kinds of radiation produced by the activated samples. During World War II the development of the atomic bomb removed the first of these two obstacles. Essential to the making of the bomb were large nuclear reactors, which generate enormous quantities of neutrons. With the early reactors built at Oak Ridge and Hanford investigators were able to analyze at least 60 trace elements in amounts as small as a millionth (10^{-6}) of a gram.

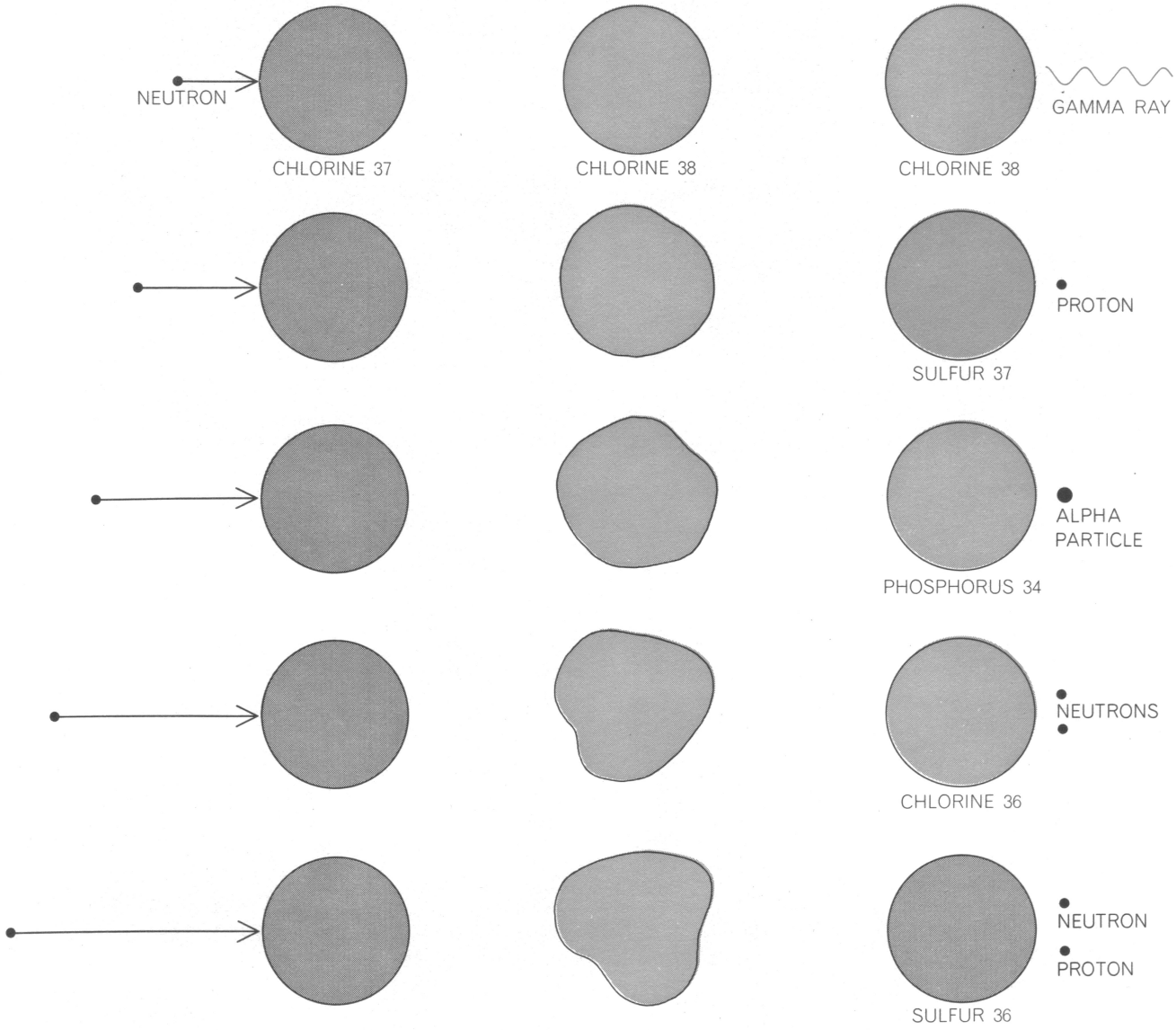
Only a few workers were able to use these facilities for conducting neutron-activation analyses; radiation-detection equipment was still crude, and many hours were required for processing the huge volume of data needed to make a single analysis. As a result neutron-activation analysis remained for many years more of a demonstrated capability than a useful analytic tool. By the middle 1950's improvements in instrumentation had significantly reduced the time required for recording and processing large quantities of data. With this final obstacle removed neutron-activation analysis can now be employed for analyzing trace elements wherever an intense source of neutrons is available.

The first step in the technique, in which the sample is exposed to the neutron beam, is called the activation step. The instability of the "compound nuclei" produced in this step arises from the immense amount of energy that is transferred to a target nucleus whenever it absorbs a neutron. Almost immediately after a compound nucleus is formed it releases its excess energy by emitting "prompt radiation"; in the process it is transformed into a nucleus that can be

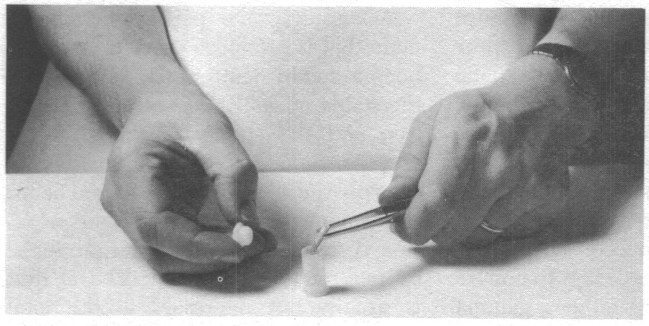
either stable or radioactive. A radioactive nucleus in turn decays and emits radiation. It is this last emission of radiation that is detected and measured in neutron-activation analysis. In addition, the prompt radiation emitted by the compound nucleus is sometimes measured to provide information about the particular reaction that takes place when a compound nucleus disintegrates.

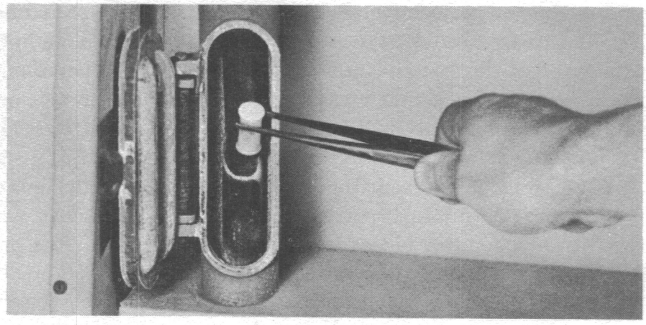
The type of nuclear reaction that will occur in this situation depends largely on the energy of the absorbed neutron. The greater the energy of the neutron, the harder it will hit the nucleus and the greater the probability is that the prompt emission will be in the form of a nuclear particle rather than an electromagnetic wave such as a gamma ray. For example, when a neutron is absorbed by a nucleus of chlorine 37 (a stable isotope of chlorine whose nucleus contains 20 neutrons and 17 protons), a highly unstable form of chlorine 38 is produced. This unstable compound nucleus decays to a more stable state by emitting either a gamma ray or one (or more) of a variety of nuclear particles, such as neutrons, protons or alpha particles [*see illustration below*].

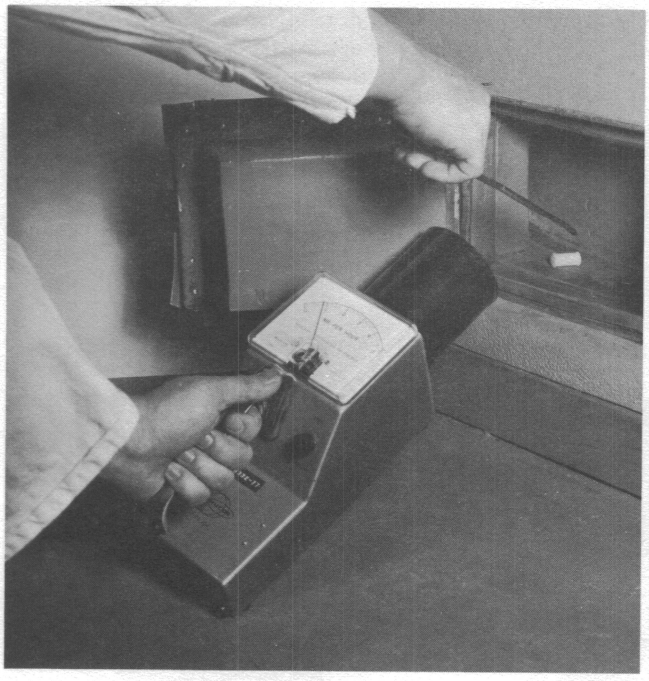
Accordingly the particular radionuclide that is produced when a sample is bombarded by neutrons depends on both the stable nucleus that absorbs the neutron and the energy of the absorbed neutron. Since a sample normally contains many different elements—and hence many more different isotopes—a profusion of radionuclides will be produced by neutron bombardment. The analyst is usually not interested in the total amount of radioactivity that is induced; he is primarily concerned with the activity of a particular radionuclide, since it is the quantitative indicator of the

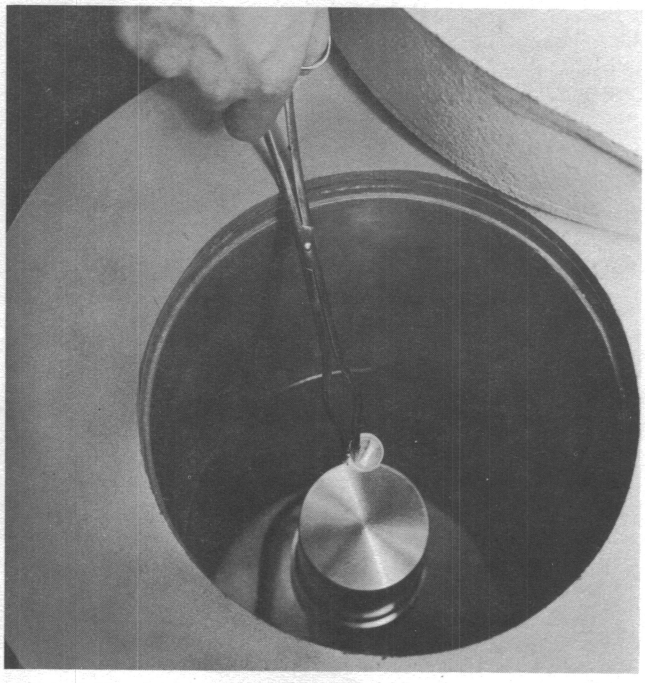


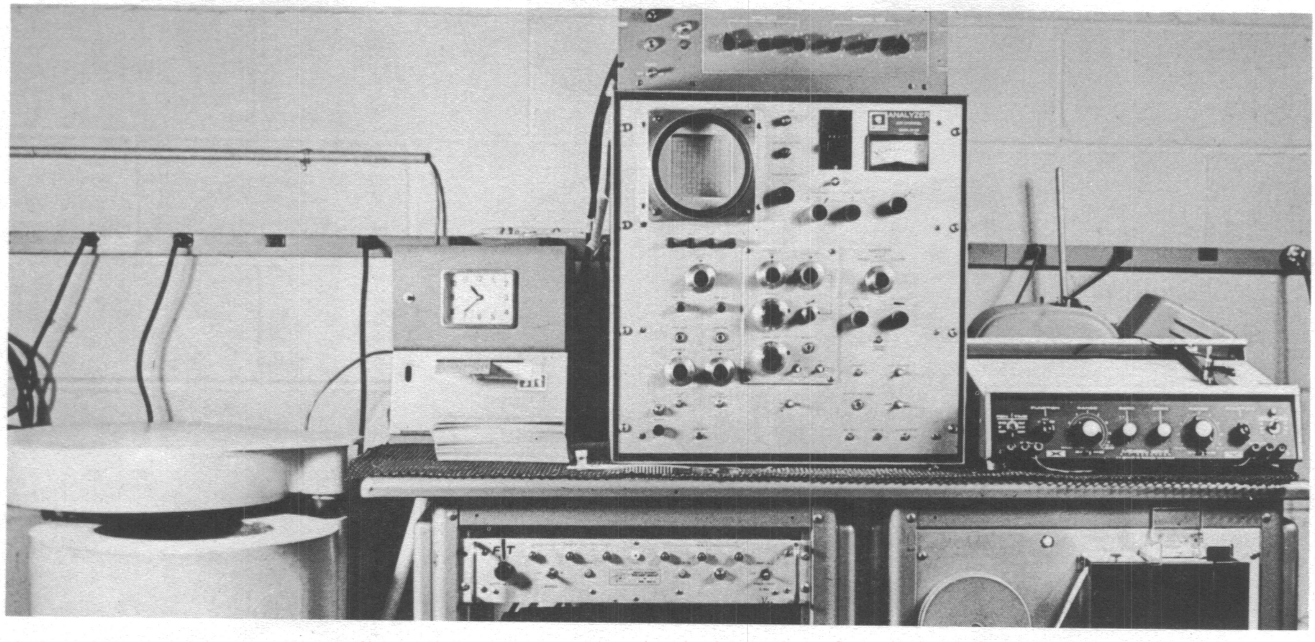
NEUTRON-ACTIVATION ANALYSIS of chlorine 37 (a stable isotope of chlorine whose nucleus contains 20 neutrons and 17 protons) begins when a chlorine 37 nucleus absorbs an extra neutron (*column 1*), producing a highly unstable "compound nucleus" of chlorine 38 (*column 2*). Almost immediately after a compound nucleus is formed it releases its excess energy by emitting "prompt radiation"; in the process it is transformed into a nucleus that can be either stable or radioactive (*column 3*). Radiation from the radioactive nuclei (*color*) can be measured. The type of nuclear reaction that will take place when a compound nucleus disintegrates depends largely on the energy of the absorbed neutron. The greater the energy of the neutron, the greater the probability that a nuclear particle rather than an electromagnetic wave will be emitted. Energy of the neutrons increases from top to bottom.











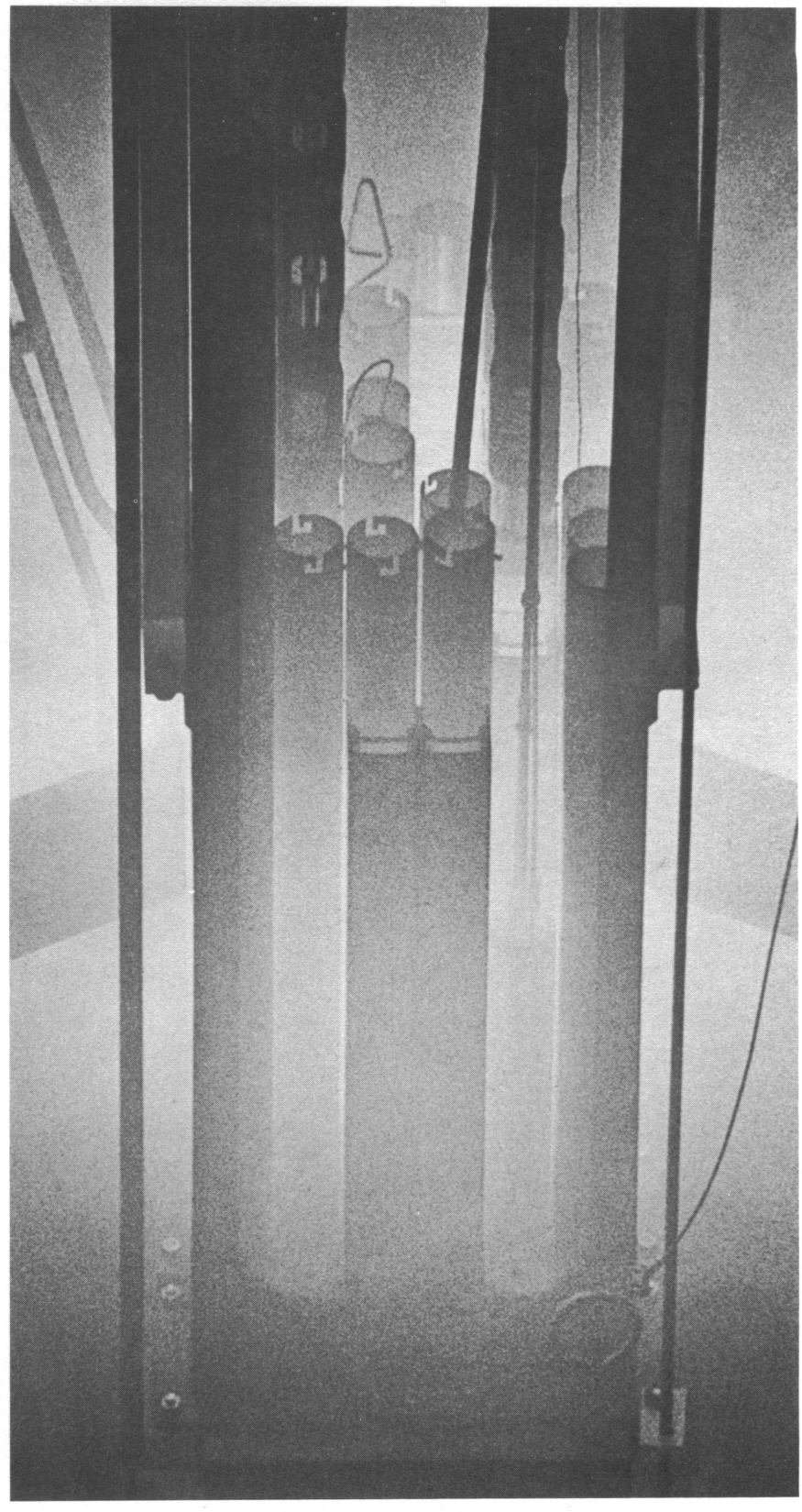
PROCEDURE followed in performing a typical neutron-activation analysis is illustrated by the sequence of photographs on this page, which were made in the authors' laboratory at the Sterling Forest Research Center of the Union Carbide Corporation. A small sample of the material to be analyzed is first inserted in a plastic irradiation container (*top left*). The container is then placed in a pneumatic transfer tube (*top right*), which leads to the core of a nuclear reactor where the sample is bombarded with neutrons. After a certain irradiation period the activated sample is returned to the laboratory (*middle left*) and placed inside a massive lead radiation shield (*middle right*). Scintillation detectors located inside the shield measure the radioactivity of the sample and transfer this information to an array of instruments (*bottom*), which analyze and record the characteristic energy spectrum of the induced radioactivity.

concentration of a particular stable isotope in the original sample.

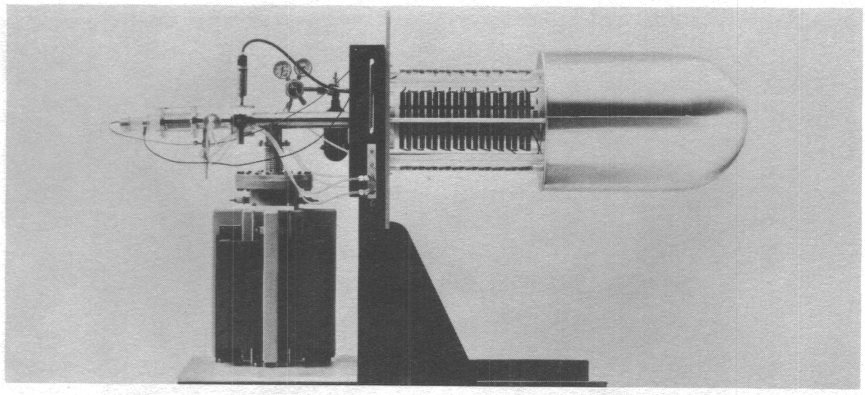
For each radionuclide the amount of radioactivity induced is proportional to the number of nuclei of the original stable isotope, the number of neutrons that are available to interact with the nuclei (the neutron flux) and the probability that a neutron will interact with a target nucleus. Every neutron that enters the sample does not interact with a nucleus, of course; the spacing between the nuclei is so large compared with both the size of the nuclei and the size of the neutrons that most of the neutrons will pass right through the sample. Only when a neutron comes fairly close to a nucleus does it have a chance to be absorbed. In somewhat oversimplified terms, the probability of an interaction between an incoming particle and a target nucleus is proportional to the apparent cross-sectional area presented by the target. According to this analogy the probability of a neutron-nucleus interaction is called the cross section of the interaction and has the dimensions of an area.

The number of radioactive nuclei in an irradiated sample also depends on how long the sample was exposed to the neutrons and on the characteristic decay rate of the radioactive nuclei, which begin to decay as soon as they are produced. The net rate of production of a radionuclide is equal to its gross production rate minus its decay rate. The radionuclide to be measured is usually not present in the original sample; consequently during irradiation the gross production rate initially far exceeds the decay rate. These two rates approach each other at an exponential rate that depends on the half-life of the radionuclide. (The half-life is the characteristic time required for half of the nuclei in a population of radioactive atoms to decay.) In time the net rate of production will be zero, and no more radioactivity will be produced. This phenomenon is called saturation.

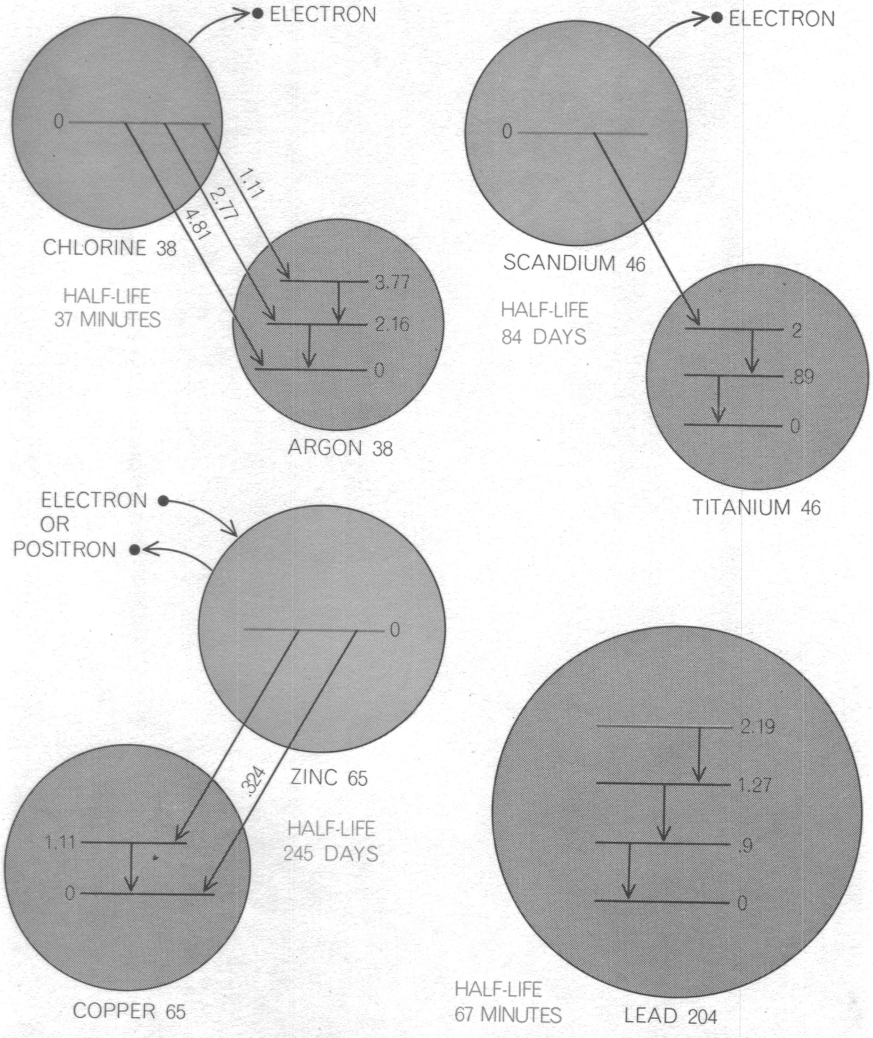
In practice the sample is exposed to neutrons long enough to produce a measurable activity of the particular "indicator" radionuclide formed by the element that is being assayed; then the sample is removed from the irradiation zone and its induced radioactivity is measured. Activation ceases when the sample is removed, and in time all the induced radioactivity will decay. Thus the amount of each radionuclide present at any time after neutron bombardment is influenced by those factors that are fixed for a given sample (the number of



NUCLEAR REACTOR is used by the authors and their colleagues as a source of neutrons for activating samples of various materials. The neutrons are emitted as a by-product of the fission of uranium 235, which takes place in the vertical pipelike fuel elements visible in the center of the photograph. The entire reactor is immersed in a large tank of water in order to slow down the energetic neutrons emitted by the fission reaction. The sample to be activated is transferred into the reactor in the vicinity of the fuel elements. The glow surrounding the fuel elements is called Cerenkov radiation; it is produced by the passage of fast electrons through the water. The photograph was made solely by the light of the Cerenkov radiation.



COCKCROFT-WALTON GENERATOR produces neutrons by bombarding a target (*inside end of tube at left*) with accelerated charged particles. The sample is placed in the path of the neutrons, which are emitted in all directions. Neutron generators produce a usable neutron flux about 10,000 times smaller than flux available from a nuclear reactor.



DECAY PROPERTIES of every radionuclide are unique to that particular nuclear species. An artificial radioactive nucleus usually decays by emitting electrons, positrons or gamma rays, or by absorbing an orbital electron. In each case the radioactive nucleus (*color*) loses internal energy, forming a more stable nucleus (*gray*), which can have the same atomic number (*arrows pointing straight down*), a higher atomic number (*arrows pointing to lower right*) or a lower atomic number (*arrows pointing to lower left*). A radioactive species can decay by a number of routes, sometimes producing a more stable species that is in an excited state. When the excited species decays to its ground state, it emits a gamma ray, which is usually associated with the decay processes of the parent radionuclide. Black numbers denote energy in millions of electron volts (MeV). Half-lives are indicated in color.

reacting isotopes, the cross section of the production reaction and the half-life of the radionuclide) and those factors that can be varied by the analyst (the neutron flux, the period of irradiation and the decay time prior to measurement of the induced activity). All these factors are expressed in a single mathematical equation called the activation equation.

There are three types of neutron source used for activating samples: nuclear reactors, neutron generators and isotopic sources. The neutron flux available in nuclear reactors allows analyses of most elements in concentrations from 10^{-12} to 10^{-6} gram. Neutron generators, such as the Cockcroft-Walton generator and the Van de Graaff generator, produce a usable flux about 10,000 times smaller than the flux available from a nuclear reactor, and this reduction in flux is accompanied by a comparable reduction in the sensitivity of measurement. The neutron yield of the isotopic sources is still lower, and they are used mainly to demonstrate the principles of the activation technique.

Since many different radionuclides are produced in a particular irradiated sample, the problem is to measure the activity of the indicator radionuclide in the presence of all the other induced radionuclides. This task is not as difficult as it sounds. It happens that every radionuclide disintegrates in a manner unique to itself. There are 66 natural radionuclides and 1,095 known radionuclides that can be artificially produced. No two of these 1,161 nuclei have exactly the same decay properties. Accordingly by measuring the decay properties of the radiation from a sample one can sometimes effectively isolate one radionuclide from all the others in the sample. When the decay properties cannot be measured directly, the radionuclides can be separated chemically. Therefore by one method or another all the activated nuclei in a sample can be identified and measured.

The radionuclides most frequently encountered in neutron-activation analysis decay by the emission of beta particles (electrons and positrons) and gamma rays. Because it is difficult to resolve beta-particle spectra resulting from mixtures of radionuclides, beta particles are not used for analysis unless they are the only emission from the indicator isotope, or unless the element can be conveniently isolated in a pure form. Gamma rays, on the other hand, are emitted with distinct energies that can be resolved by the standard technique known as scintilla-

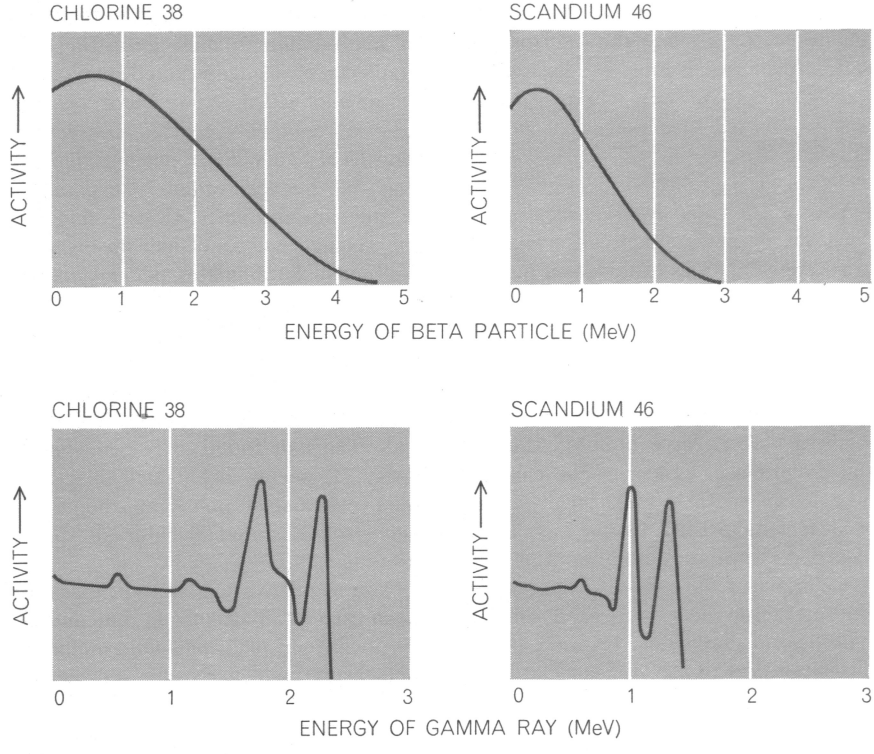
tion spectrometry; as a result most neutron-activation analyses are based on gamma ray data.

In scintillation spectrometry gamma rays are detected by causing them to interact with a material that responds to the interaction by scintillating, that is, by emitting photons of visible light whose intensity is directly proportional to the energy of the absorbed gamma ray. The most widely used scintillating material is a single crystal of sodium iodide "doped" with a small amount of the element thallium. The crystal is coupled to a photomultiplier tube, which detects the light emissions and converts them into electronic pulses. These pulses are fed into a multichannel pulse-height analyzer, which sorts them according to amplitude and counts the number of pulses in each amplitude range during a given counting period. The result is a record of the energy distribution of the gamma rays emitted by the radioactive nuclei in the sample.

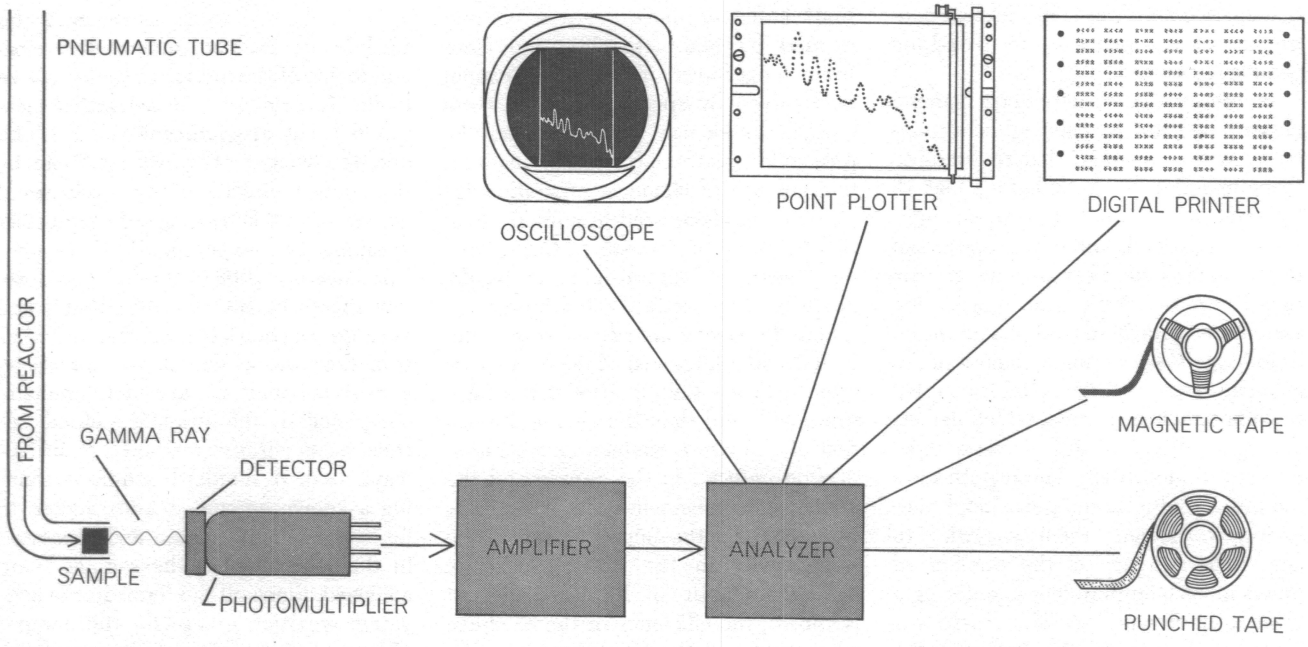
In some cases the gamma rays emitted by different radionuclides are so similar in energy that they cannot be adequately resolved in the gamma ray spectrum. In these instances the different characteristic decay rates of the individual radionuclides can be used to isolate one isotope from another. It is often possible to maximize or minimize the activity of one radionuclide with respect to another in an activated sample by properly adjusting the duration of the irradiation period and the decay period. The use of time as an independent variable is unique to the neutron-activation technique, giving the analyst two additional factors to manipulate to his advantage. The ability to discriminate in this manner depends primarily on the difference in the half-lives of the radionuclides induced in the samples. Short irradiation periods are used to maximize the ratio of short-lived nuclei with respect to longer-lived nuclei when the longer-lived ones may give rise to interfering radiations. By the same token longer irradiation periods are employed to maximize the long-lived nuclei, and the analyses for these nuclei are completed after the short-lived nuclei have been allowed to decay.

When the radioactivity of the indicator nuclide cannot be accurately measured in the presence of other radionuclides, even after the time variables are taken into account, chemical methods are used to isolate the individual radionuclides. This situation usually obtains when the radiations from the desired indicator nuclide are effectively masked by more abundant radiations from longer-lived nuclei and cannot be resolved by spectral analysis. Even though the element of interest may be present in extremely small quantity, the methods of chemical separation are the same as those used in normal quantitative analyses. Instead of the separations' being carried out on a microscale, however, they are facilitated by the addition of a known amount (approximately 10 to 50 milligrams) of the element of interest to the sample after it has been irradiated, and then the chemical isolation of a mixture containing the radionuclide induced in the sample and the added stable element. The analysis is completed by the measurement of the radioactivity in the isolated mixture. Since the quantity of the added element is known, the efficiency of the chemical separation is easily determined, and the radioactivity measurements can be corrected for any losses caused by chemical processing after irradiation. Since the radiochemical separation need not be quantitative, the separation procedures can be shortened and the tedious work frequently required for quantitative analysis can be minimized.

Once the radiations of the indicator nuclide are isolated, quantitative measurements of the induced activity can be made. In principle the activation technique is an absolute analytic method, and the weight of an element can be determined directly from the measured activity and a knowledge of the nuclear constants and experimental conditions. But since it is difficult to make an absolute measurement of radioactivity, and because reaction cross section and neutron flux are seldom known precisely, activation analyses are not normally completed by the absolute method. Instead a comparative method is almost always used. A standard sample containing a known amount of the element to be analyzed is irradiated and processed in the same way as the sample being analyzed. Since all the terms in the activation equation except for the number of target atoms and the induced activity are the same for the control and the test sample, the activity of the indicator nuclide in the test sample can be directly compared with the activity of the control to obtain the weight ratio of the desired element in two samples. Analyses by this method can be completed with an accuracy of about 2 percent of the actual



MOST RADIONUCLIDES DECAY by emitting beta particles (electrons and positrons) or gamma rays. The beta-particle spectra of low-energy beta-particle emitters (*top*) are difficult to resolve in the presence of high-energy emitters; as a consequence beta particles are rarely used for neutron-activation analysis. Gamma rays, on the other hand, are emitted with distinct energies that can be readily resolved by scintillation spectrometry (*see illustration on page 8*); as a result most neutron-activation analyses use gamma ray data.



GAMMA RAYS from an activated sample are measured by allowing them to fall on a scintillation detector, which emits photons of visible light whose intensity is directly proportional to the energy of the absorbed gamma ray. The detector is coupled to a photomultiplier tube, which detects the light emissions and converts them into electronic pulses. The pulses are amplified before being fed into a multichannel pulse-height analyzer, which sorts them into a record of the energy distribution of the original gamma rays. The gamma ray data can be "read out" of the analyzer in a number of ways, some of which are represented schematically in illustration.

concentration at levels down to as little as 10 times the lower limit of detection. Analyses with an accuracy of better than 1 percent of the actual value are possible but extremely difficult to attain by this technique. As a result the neutron-activation technique is most widely used for trace-element analysis where an accuracy of from 2 to 5 percent is permissible.

One of the most fascinating applications of neutron-activation analysis in recent years was undertaken in 1961 by two Swedish physicians, Sten Forshufvud and Anders Wassén, in collaboration with Hamilton Smith of the Department of Forensic Medicine at the University of Glasgow. For a long time medical writers have doubted the official diagnosis of cancer as the cause of Napoleon's death in exile on the island of St. Helena in 1821. On the basis of the recorded symptoms a number of alternative diagnoses have been proposed, including peptic ulcer, malaria, dysentery, syphilis and gonorrhea. In order to test the hypothesis that Napoleon had in reality been poisoned, Forshufvud and his co-workers obtained well-documented samples of Napoleon's hair, which was shaved from his head the night after he died, apparently to provide as much hair as possible for distribution as souvenirs and to facilitate the making of a death mask. Several of the samples were irradiated with neutrons in a nuclear reactor. Consecutive half-millimeter lengths of rather long hairs were then analyzed for the presence of arsenic. The concentration of arsenic in the segments was found to be much higher than that in normal human hair, indicating that Napoleon may have suffered from chronic arsenic intoxication. Moreover, the arsenic content of the consecutive lengths of hair was not uniform, indicating that he was probably exposed to arsenic intermittently. Considering that the average growth of hair on the scalp is about .35 millimeter per day, it was possible to infer that periods of higher arsenic concentration in the hair coincide with days on which Napoleon was reported to be sicker during his captivity. Hence, judging by the arsenic concentration in hair found in healthy people today, it seems likely that Napoleon died from arsenic poisoning, the arsenic being administered at intervals during his illness.

Neutron-activation analysis has also been used in an attempt to date and authenticate old paintings. One method is to measure the concentrations of various trace elements in white lead paint, which has been used as a primer and white pigment since the Middle Ages. The purity of white lead is directly related to the purity of the lead used to make the paint. Since the metallurgical techniques for purifying lead have been modified several times during the past five centuries, the trace-element content of the white lead in a particular painting can be used to estimate the age of the painting. Using the neutron-activation technique, Jan Houtman and J. Turkstra of the Reactor Instituut at Delft in the Netherlands have analyzed tiny samples of white lead removed from a number of authentic "old masters" at museums in Belgium and the Netherlands [*see illustration on page 10*].

They have found that the concentrations of copper, silver, mercury and manganese were fairly high and constant in white lead made between 1500 and about 1850; after 1850 these concentrations dropped to about a tenth of their earlier values and have remained low and constant ever since. Similarly, the concentration of chromium was found to be high before 1650 and much lower since 1650. On the other hand, the concentrations of zinc and antimony, which were quite low before 1940, have increased considerably in recent years. The great sensitivity of the neutron-activation technique makes possible a complete analysis using less than a milligram sample of the white lead, which can be obtained from the edge of the painting without causing any noticeable damage. Data obtained by the neutron-activation technique can be used not only to date a painting but also in some cases to determine the identity of the artist by comparing the paint with the paint of authenticated works.

Most of the trace-element analyses

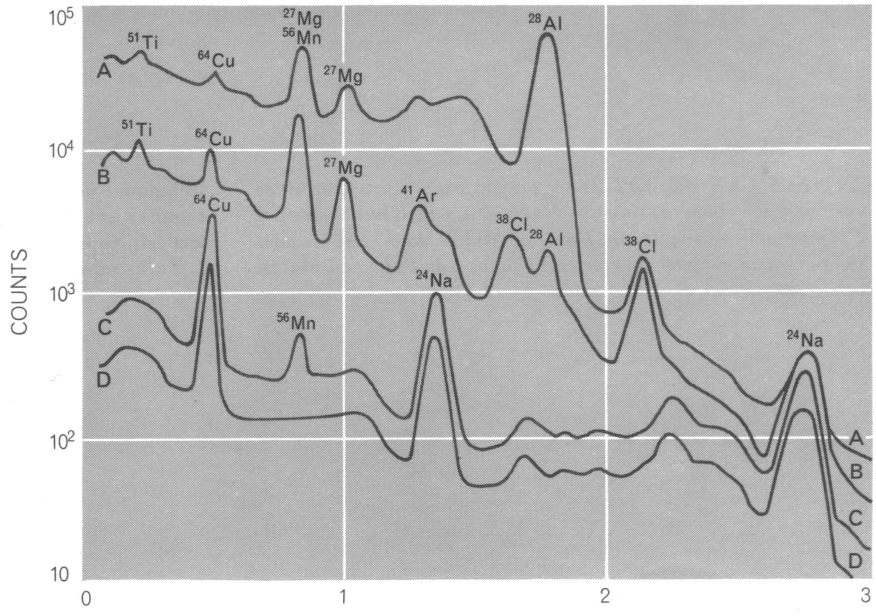
performed by the neutron-activation technique are of course more mundane than the ones just described. Nonetheless, the technique has played a key role in solving many important technological problems. For example, it has been used in the development of purer materials for manufacturing transistors, high-strength metal alloys and plastics. One noteworthy problem that was solved had to do with certain epoxy resins, which originally had a short and unpredictable shelf life. A neutron-activation analysis of these resins revealed the presence of very small amounts of a number of metals. When these trace contaminants were removed, the shelf life of the resins was extended considerably. Apparently the trace contaminants had catalyzed the fixing of the resin.

Neutron-activation techniques are also used to characterize raw materials by their trace-element content. In one of our studies at Union Carbide we discovered that the trace-element content of the mineral chromite in ore deposits is constant within a given deposit but is quite different from that found in other deposits. This information is useful for measuring the extent of a given deposit and for determining why one source of ore gives rise to a better product than another. Petroleum geologists, using a similar approach, employ trace-metal analysis to determine the extent of oil reserves, the stratigraphic position of oil traps, the age of crude oils and the fundamental principles governing the origin, migration and alteration of petroleum. Soils from widely separated locations also show large variations in trace-element concentrations; as a result vegetation exhibits trace-element characteristics peculiar to the area in which it grows. Such information is used by geologists to locate new ore deposits, by ecologists to study the relation between the growth of certain trees and shrubs and their environment, and by official investigators to identify the original source of raw opium that has been confiscated.

The neutron-activation technique has developed into one of the most sensitive, accurate and broadly applicable methods for the measurement of minor and trace elements in biological specimens. It is being used to elucidate the role of trace elements in normal and abnormal physiological functions. Typical subjects of study include selenium in "white muscle" disease, manganese in heart disease, arsenic and molybdenum in uremia, copper and manganese in leukemia and zinc in the formation of kidney stones. Cadmium, mercury and uranium (in addition to arsenic) are analyzed in biological samples to evaluate evidence of poisoning. Chronic poisoning is distinguished from acute poisoning by analyzing blood, urine, stomach fluids, fingernail clippings and hair. Abnormal concentrations of poisons in all these substances indicate chronic, or long-term, exposure; abnormal concentrations in all but the fingernails and hair indicate acute exposure, since it takes time for the hair and fingernails to grow.

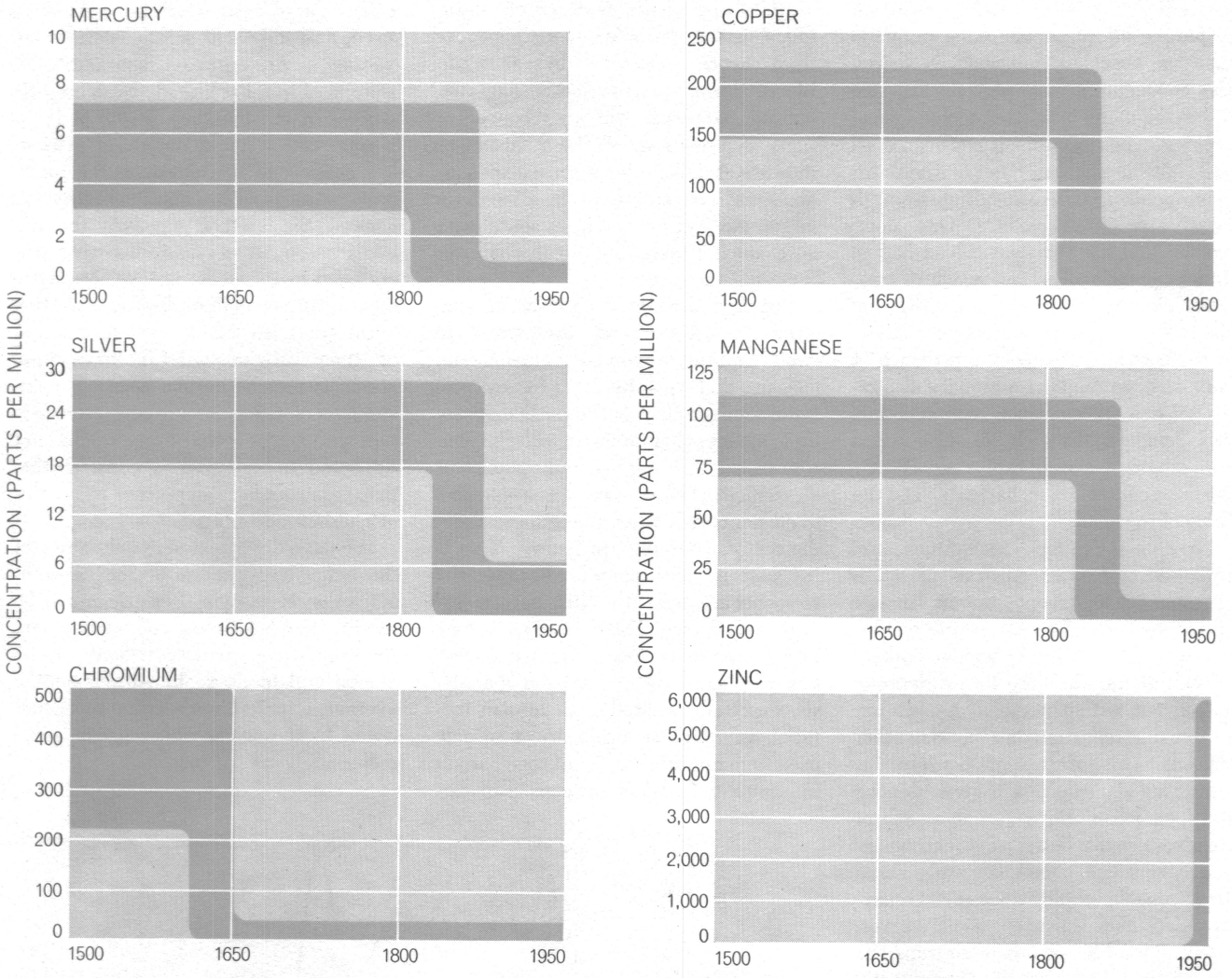
Although the neutron-activation technique is used almost exclusively to detect various chemical elements and measure their quantity, it is basically an isotopic method; it is the particular isotopic nucleus that interacts with the neutron to produce a given radioisotope of an element. The technique is applicable to elements only when the isotopic abundances of a sample are known. This is the case in most samples, because the elements are present in their natural isotopic abundance. The technique is now being used with increasing frequency, however, as an isotopic method of analysis, especially in studies of human metabolism. Radioisotopes are routinely used in diagnostic studies of the changes in normal body functions caused by disease. Nonradioactive isotopes can also be used, and indeed they have certain advantages. An enriched nonradioactive isotope of iron has already been used for studies of the function of iron in blood plasma and a stable isotope of calcium has been used to determine the metabolic pathway of calcium in children. In general the method involves the administration of a nonradioactive salt enriched in a specific isotope. Measurement of the change in the concentration of the administered isotope over a period of time provides useful information about its metabolic role. The major advantage of using a nonradioactive isotope as a tracer is that the patient is not subjected to radiation, a consideration that is particularly important in studies of children and pregnant women.

We have described here only a few of the many applications of the neutron-activation technique. Because it can be used to analyze samples of virtually any size for their content of at least 70 elements, and to do so to within, on the average, a billionth of a gram, this product of basic nuclear research will be an increasingly useful tool.



RADIONUCLIDE	HALF-LIFE	RADIONUCLIDE	HALF-LIFE
ALUMINUM 28 (^{28}Al)	2.3 MINUTES	ARGON 41 (^{41}Ar)	1.83 HOURS
TITANIUM 51 (^{51}Ti)	5.8 MINUTES	MANGANESE 56 (^{56}Mn)	2.58 HOURS
MAGNESIUM 27 (^{27}Mg)	9.5 MINUTES	COPPER 64 (^{64}Cu)	12.9 HOURS
CHLORINE 38 (^{38}Cl)	37.3 MINUTES	SODIUM 24 (^{24}Na)	15 HOURS

SIMULTANEOUS ANALYSIS of a number of elements in a sample is possible using their gamma ray spectra. The spectra shown here were recorded for a neutron-irradiated piece of paper after decay periods of three minutes (*A*), 10 minutes (*B*), four hours (*C*) and 24 hours (*D*). The peaks in the spectra can be identified by referring to the key at bottom. Because of the different half-lives of the elements, the analysis of a particular element can be facilitated by choosing a decay time when the emissions from the "indicator" radionuclide are least interfered with by the emissions from other radionuclides in the sample.



PAINTINGS CAN BE DATED by measuring the concentrations of various trace elements in white lead paint, which has been used as a primer and white pigment since the Middle Ages. The changes in trace-element content are related to changes in the metallurgical techniques used to purify lead. The neutron-activation technique requires less than a milligram of white lead to perform a complete analysis. Graphs are based on data obtained by Jan Houtman and J. Turkstra of the Reactor Instituut at Delft in the Netherlands.

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